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RADIATION QUALITY ASSURANCE INTERCOMPARISON STUDIES 1974-1975

Environmental Monitoring and Support Laboratory
Las Vegas, Nevada

October 1975

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16. ABSTRACT

The Quality Assurance Branch of the Monitoring Systems Research and Development Division at the U.S. Environmental Protection Agency's Environmental Monitoring and Support Laboratory-Las Vegas prepares and distributes calibrated low-level radioactive solutions to Federal, State, and private laboratories involved in environmental radiation monitoring and surveillance. These solutions are utilized for both the calibration of counting instruments and chemical yield determinations. Since the laboratories using these samples must have confidence in their accuracy, the Quality Assurance Branch has instituted intercomparison studies with the National Bureau of Standards and with the Energy Research and Development Administration Health and Service Laboratory. The results of the studies conducted during 1974 and 1975 are described.

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RADIATION QUALITY ASSURANCE INTERCOMPARISON STUDIES 1974-1975

by

Quality Assurance Branch
Monitoring Systems Research and Development Division
Environmental Monitoring and Support Laboratory
Las Vegas, Nevada 89114

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U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
LAS VEGAS, NEVADA 89114

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Effective June 29, 1975, the National Environmental Research Center-Las Vegas (NERC-LV) was designated the Environmental Monitoring and Support Laboratory-Las Vegas (EMSL-LV). This Laboratory is one of three Environmental Monitoring and Support Laboratories of the Office of Monitoring and Technical Support in the U.S. Environmental Protection Agency's Office of Research and Development.

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RADIATION QUALITY ASSURANCE INTERCOMPARISON STUDIES
1974-1975

INTRODUCTION

The Quality Assurance Branch of the Technical Support Laboratory at the U.S. Environmental Protection Agency's (EPA) National Environmental Research Center-Las Vegas (NERC-LV) prepares and distributes calibrated low-level radioactive solutions to federal, state, and private laboratories involved in environmental radiation monitoring and surveillance. These solutions are utilized for both the calibration of counting instruments and chemical yield determinations. Since the laboratories using these samples must have confidence in their accuracy, the QA Branch has instituted a continuing intercomparison studies program with the National Bureau of Standards (NBS), the goal of these studies being to establish the traceability of the EPA's calibrated radiation samples to the NBS.

The expression "traceability to NBS," although appearing frequently in guidelines and procedures referring to the measurement of radioactive materials, has not been adequately defined in the guidelines and appears to be a much misunderstood term. In many areas of metrology, traceability to the NBS can be readily established in a direct manner. A standard is obtained from the NBS and, by using the NBS prescribed procedures, the instrument of interest is calibrated and its precision and accuracy documented. For many measurements (e.g., voltage measurements using standard cells) such a procedure is sufficient to establish traceability to the NBS. However, since the activity of all radionuclides decreases with time, it is not possible to duplicate a measurement of the same material at different times. Therefore, the measurement of radioactive materials and the establishment of their traceability to

the NBS require a different approach. Moreover, many gaps still exist in our knowledge concerning the half-lives and decay schemes of many radionuclides. Consequently, to obtain traceability to the NBS in the measurement of radionuclide activity each radionuclide must be assayed on an individual basis. Cavallo, *et al.* (1), have defined the expression "traceable to NBS" and indicated how both direct and indirect traceability to NBS may be obtained.

> "Direct traceability to the national radioactivity measurements system (NRMS) exists when any 'outside' laboratory prepares a batch of calibrated radioactivity standards and submits several randomly selected samples to the national standardizing laboratory for confirmation and verification.... Indirect traceability to the NRMS exists when the national laboratory provides 'unknown' calibrated radioactivity samples to one or more measurement laboratories, who in turn make measurements of activity that agree within certain specified limits with those of the national laboratory. Thus we can have 1%, 5%, etc., traceability.... Indirect traceability only means that the 'lower level' laboratory has the potential to make measurements that are consistent within the NRMS...."

Addressing the question of using standards in establishing traceability, Cavallo, *et al.* (1), state:

> "If an 'outside' laboratory regularly uses NBS radioactivity standards to calibrate its measuring equipment, this does not, in our view, constitute traceability. Only when that laboratory can measure the activity of an unknown sample and send back values that agree with our values within a certain specified range of error do we consider

that traceability has been established. And that condition can be achieved without using a single NBS radioactivity standard."

PROCEDURES

Utilizing the approach suggested by Cavallo, *et al.* (1), the Quality Assurance Branch, NERC-LV, initiated a program designed to establish both "direct" and "indirect" traceability of radioactive nuclides to the NBS within plus or minus 5 percent at the 1 sigma confidence level. NBS personnel reviewed the objectives of the EPA's calibrated sample distribution program, inspected the laboratory, facilities, and instrumentation available at the NERC-LV, then outlined the program required to establish the desired 5 percent traceability.

The first phase of the program involved direct traceability studies wherein radioactive solutions, prepared and calibrated by the QA staff, were submitted to the NBS for their measurements. The samples submitted were 5-milliliter (ml) aqueous solutions of manganese-54, zinc-65, strontium-89, strontium-90, and cobalt-60 with activity levels ranging from 237 disintegrations per minute per gram (dps/g) to 2210 dps/g. These samples were selected at random from the inventory of the EPA's calibrated sample distribution program.

The second phase of the program, an indirect traceability study, required that radioactive solutions prepared by the NBS be measured by the QA staff. Since the initiation of this phase of the study, the NBS has submitted 11 different radionuclides for analysis. These include strontium-89, strontium-90, mercury-203, chromium-51, cesium-137, cadmium-109, selenium-75, carbon-14, iron-59, silver-110m, and cesium-134. Measurements have been completed and the results reported to NBS for strontium-89, strontium-90, mercury-203, chromium-51, cesium-137, cadmium-109, and selenium-75. Work is currently in progress on carbon-14, iron-59, silver-110m, and cesium-134.

In addition to the NBS traceability studies, the Quality Assurance staff has participated in intercomparison studies with the Energy Research and Development Administration's (ERDA) Health Services Laboratory (HSL) at Idaho Falls, Idaho. These studies were deemed necessary since the EPA's radiation quality assurance program involves the participation of federal, state, and private laboratories, many of which also participate in the quality control programs of ERDA. It is, therefore, essential that the measurements being made and the calibrated samples being distributed by the two Federal agencies involved in environmental radiation monitoring be in close agreement. To ascertain whether such agreement exists, calibrated samples were exchanged by the two laboratories. The Quality Assurance Branch, NERC-LV, submitted calibrated samples of 15 different radionuclides for measurement by the HSL and the HSL submitted 8 different radioactive solutions to the NERC-LV for analysis.

The measurement of the samples supplied by the NBS requires, in addition to the measurement of activity, a qualitative and quantitative analysis of radionuclide impurities and a rigorous analysis of errors. A questionnaire, typical of those which accompany each NBS sample, is shown in the Appendix.

The counting instruments utilized for the routine calibration of low-level radioactive samples and for the traceability studies with the NBS include:

1. A 4-inch by 4-inch NaI well crystal coupled to a 400 channel Technical Measurements Corporation Model 404c multichannel analyzer. (Figure 1)
2. A 16% efficient Ge(Li) coaxial detector coupled to a Nuclear Data 4096 channel analyzer using an Ortec Model 452 linear amplifier. (Figure 2)
3. A Beckman LS-100 liquid scintillation counter.

The measurement of the gamma emitting radionuclides in the indirect traceability studies involved the determination of the total activity of

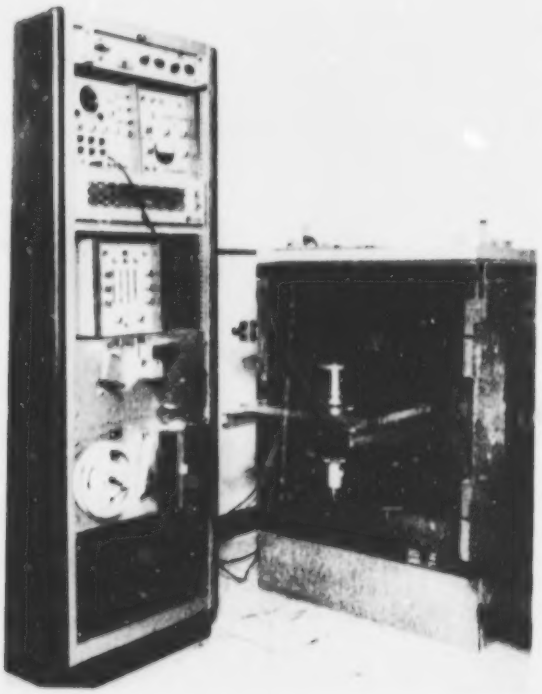

Figure 1. 4-inch by 4-inch NaI Well Crystal Coupled to a Multichannel Analyzer

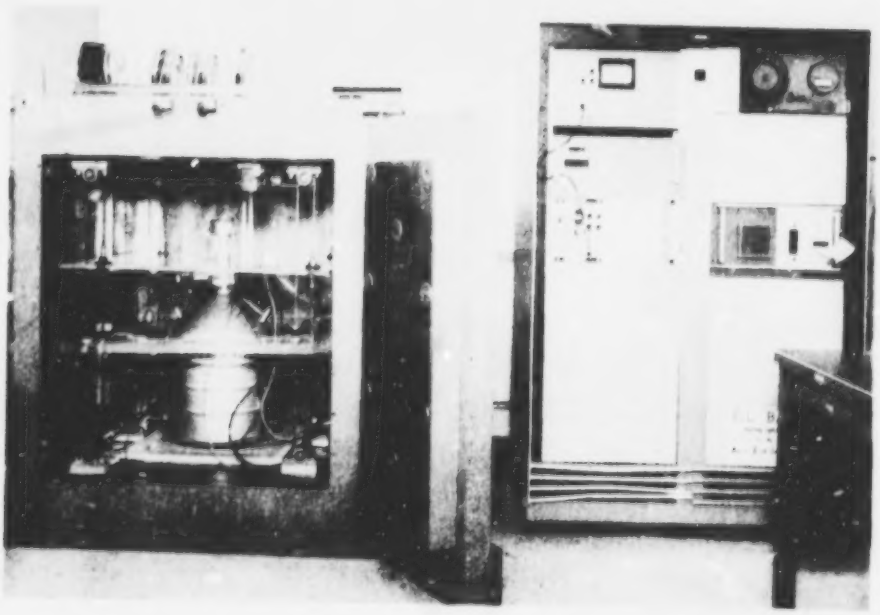

Figure 2. Ge(Li) Coaxial Detector Coupled to a Channel Analyzer Using a Linear Amplifier

the ampul supplied by the NBS and the activity per gram of solution. The selection of the counting instrument(s) and the method(s) of analysis used for the studies were made on an individual basis.

The measurements of cesium-137, cadmium-109, and selenium-75 were done on the Ge(Li) system while chromium-51 and mercury-203 were measured using a NaI system. Determination of the total activity per ampul was accomplished by making replicate measurements of each of the ampuls supplied by the NBS. To determine the activity per gram of solution, each NBS ampul was opened, diluted, and 5-ml aliquots of the dilution prepared and counted. The gamma emission rate of each of these aliquots was then determined by comparing their gamma-emission rates with those of the appropriate standard(s).

For cesium-137 an NBS cesium-137 standard, calibrated for emission rate, was utilized. The activity of the chromium-51 sample was determined using a chromium-51 standard (Amersham-Searle) calibrated for activity. In the case of cadmium-109, a cadmium-109 standard (Laboratoire de Metrologie des Rayonnements Ionisants), calibrated for activity, as well as an NBS mixed radionuclide standard, calibrated for emission rate, was employed. The gamma emission rate of mercury-203 was determined using a mercury-203 standard (Amersham-Searle) calibrated for activity. The activity of selenium-75 was determined by comparing the gamma emission rate using a mercury-203 standard (Amersham-Searle), an NBS cobalt-57 standard, and an aliquot of an Amersham-Searle cobalt-57 standard, all of which were calibrated for activity.

In addition to the gamma-emitting samples, a sample containing a mixture of strontium-89 and strontium-90 was submitted by the NBS. The activities per gram of solution for both strontium-89 and strontium-90 were measured on a Beckman LS-100 liquid scintillation counter. The LS-100 was standardized using strontium-89 and strontium-90 standards obtained from Amersham-Searle. The Cerenkov method of counting was employed both for standardization and sample analysis of strontium-89 and yttrium-90 (2). The unknown strontium-89 and strontium-90 mixture was received from the NBS in a sealed glass ampul. The ampul was opened

and the contents transferred to a tared 100-ml volumetric flask. The flask and sample were weighed, diluted using strontium-yttrium carrier in 0.1N nitric acid solution and reweighed.

Five-tenths gram portions of the unknown NBS sample and the strontium-89-90 mixture were measured using the LS-100. Five-tenths gram portions of a known yttrium-88 and 0.5 ml of a known strontium-85 sample were measured using a NaI well counter. The yttrium-88 and strontium-85 were used for yield determination. The yttrium was precipitated as a hydroxide three successive times for separation of the strontium from the yttrium. The yttrium-88 was then counted using the well counter and the yttrium-90 (of the known and NBS unknown) was counted using the LS-100. The supernate of the NBS known and strontium-85 samples were evaporated to approximately 10 ml and fuming nitric acid was added to drop out the strontium nitrate. This step was repeated. The strontium-85 was then counted in the well counter and the strontium-89 in the LS-100. The counting efficiency using Cerenkov radiation was 51 percent for yttrium-90, 0.1 percent for strontium-90, and 26 percent for strontium-89.

The procedures used in the preparation and measurement of the EPA's calibrated radioactive samples which were submitted to the NBS in the direct traceability studies, and to HSL in the intercomparison study, have been previously described (3).

RESULTS

The results of the direct traceability studies are summarized in Table 1. These data indicate that the measurements of the five radionuclide solutions prepared by the EPA's QA staff, NERC-LV, and submitted to the NBS for assay were well within the desired plus or minus 5%. In no case did the difference between the EPA's values and those of the NBS [(NBS-EPA)/NBS x 100] exceed 2.4%.

Table 1. RESULTS OF DIRECT TRACEABILITY STUDIES

Nuclide	EPA Measured Activity (dps/g)	NBS Measured Activity (dps/g)	EPA/NBS
^{60}Co	2210 ± 6%*	2196 ± 1.3%*	1.006
^{54}Mn	2059 ± 9%	2066 ± 2.7%	.997
^{65}Zn	2200 ± 9%	2213 ± 2.3%	.994
^{89}Sr	237 ± 9%	239.2 ± 3.6%	.989
^{90}Sr	1179.3 ± 9%	1152 ± 2.5%	1.024

* Total error

A comparison of the data obtained by the EPA with that of the NBS in the indirect traceability studies is shown in Table 2. The measurements of the NBS solutions made by the EPA are in good agreement with those of the NBS as indicated by the EPA/NBS ratios. With the exception of selenium-75, the differences between NBS measurements and EPA measurements range from minus 2.8 percent to plus 3 percent for cadmium-109.

Table 2. RESULTS OF INDIRECT TRACEABILITY STUDIES

Nuclide	EPA Measured Activity[a]	NBS Measured Activity[a]	EPA/NBS
^{109}Cd	$1.16\times10^{5}\gamma s^{-1}g^{-1}\pm5.6\%$ $1.23\times10^{5}\gamma s^{-1}g^{-1}\pm1.5\%$[b]	$1.196\times10^{5}\gamma s^{-1}g^{-1}\pm1.88\%$	.970 1.028
^{75}Se	$36\mu Ci\ g^{-1}$ +20% -15% $189\mu Ci\pm11\%$	$39.19\pm1.03\mu Ci\ g^{-1}$ $199.36\mu Ci\pm2.6\%$	.919 .951
^{89}Sr	$90.0_8 nts^{-1}g^{-1}\pm12\%$	$90.3_6 nts^{-1}g^{-1}\pm3\%$	.997
^{90}Sr	$5.17 nts^{-1}g^{-1}\pm3\%$	$5.1 nts^{-1}g^{-1}\pm3\%$	1.00
^{203}Hg	$85.1\mu Ci\ g^{-1}\pm13\%$	$83.14\mu Ci\ g^{-1}\pm1\%$	1.024
^{51}Cr	$32.3\mu Ci\ g^{-1}\pm4.27\%$	$31.81\mu Ci\ g^{-1}\pm1.5\%$	1.015
^{137}Cs	$1.22\mu Ci\ g^{-1}\pm2.4\%$	$1.24\mu Ci\ g^{-1}\pm2.7\%$	.981

a Uncertainties at 99.7% CL
b From total activity of source

The data obtained from the interchange of samples between the NERC-LV and HSL are summarized in Tables 3 and 4. As with the NBS studies, there is generally good agreement between the measurements of the 16 different radionuclides made by the two laboratories.

Table 3. RESULTS OF INTERCOMPARISON STUDIES WITH THE HSL (Sample Prepared by EPA - Measured by HSL)

Nuclide	EPA Measured Activity (dpm g^{-1})	HSL Measured Activity (dpm g^{-1})	EPA/HSL
^{22}Na	1.73×10^4	$1.69 \pm 0.04 \times 10^4$	1.03
^{46}Sc	4.31×10^4	$4.30 \pm 0.05 \times 10^4$	1.00
^{54}Mn	1.81×10^4	$1.91 \pm 0.02 \times 10^4$	0.95
^{58}Co	1.38×10^4	$1.38 \pm 0.01 \times 10^4$	1.00
^{60}Co	2.47×10^4	$2.47 \pm 0.04 \times 10^4$	1.00
^{63}Ni	3.51×10^4	$3.37 \pm 0.03 \times 10^4$	1.04
^{65}Zn	1.81×10^4	$1.91 \pm 0.04 \times 10^4$	0.95
^{85}Sr	7.92×10^3	$8.07 \pm 0.07 \times 10^3$	0.98
^{89}Sr	4.77×10^3	$4.66 \pm 0.09 \times 10^3$	1.02
^{90}Sr	1.14×10^5	$1.17 \pm 0.02 \times 10^5$	0.97
^{103}Ru	1.37×10^4	$1.40 \pm 0.02 \times 10^4$	0.98
^{106}Ru	2.59×10^4	$2.45 \pm 0.07 \times 10^4$	1.06
^{124}Sb	5.57×10^4	$5.27 \pm 0.04 \times 10^4$	1.06
^{137}Cs	3.81×10^4	$3.90 \pm 0.01 \times 10^4$	0.98
^{140}Ba	2.07×10^4	$2.03 \pm 0.03 \times 10^4$	1.02

Uncertainties at $\pm 1\sigma$ CL

Table 4. RESULTS OF INTERCOMPARISON STUDIES WITH THE HSL
(Samples Prepared by HSL - Measured by EPA)

Nuclide	EPA Measured Activity ($dpm\ g^{-1}$)	HSL Measured Activity ($dpm\ g^{-1}$)	EPA/HSL
^{137}Cs	$3.44 \times 10^4 \pm 3\%$	3.44×10^4	1.00
^{54}Mn	$4.88 \times 10^4 \pm 3\%$	4.99×10^4	.978
^{65}Zn	$1.02 \times 10^4 \pm 4\%$	1.08×10^4	.944
^{88}Y	$3.09 \times 10^4 \pm 2\%$	3.11×10^4	.994
^{60}Co	$3.48 \times 10^4 \pm 2\%$	3.54×10^4	.983
^{90}Sr	$2.10 \pm 0.02 \times 10^4$	$2.26 \pm 0.05 \times 10^4$	.929
^{89}Sr*	$3.43 \pm 0.08 \times 10^4$	$4.92 \pm 0.07 \times 10^4$	*

Uncertainties at 1σ CL

* $+5.18 \times 10^3$ strontium-90 contamination.

SUMMARY AND CONCLUSIONS

Direct and indirect traceability studies with the NBS, as well as intercomparison studies with the HSL, have been conducted by the EPA's Quality Assurance Branch, NERC-LV. Direct traceability studies have been made for five radionuclides: manganese-54, cobalt-60, zinc-65, strontium-89, and strontium-90. In addition, indirect traceability studies with the NBS have been made for seven radionuclides: chromium-51, selenium-75, strontium-89, strontium-90, cadmium-109, cesium-137, and mercury-203. As indicated by EPA/NBS ratios, all of the EPA measurements, with the exception of the activity per unit mass determination of selenium-75, have agreed with those of the NBS within the desired plus or minus 5 percent.

Intercomparison studies, involving the exchange and measurement of calibrated radioactive samples by the HSL and the NERC-LV indicate good agreement between the two laboratories. With the exception of zinc-65 and strontium-90, the measurements of the two laboratories do not differ by more than plus or minus 5 percent.

These results indicate that the QA Branch, NERC-LV, has the facilities, instruments, staff, and expertise required to measure aqueous solutions of nine different radionuclides to within plus or minus 5 percent of the NBS values. The traceability studies with NBS are on a continuing basis. Measurements of four additional radionuclide solutions prepared by NBS (carbon-14, iron-59, silver-110m, and cesium-134) are currently in progress.

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APPENDIX. QUESTIONNAIRE SUPPLIED WITH UNKNOWN SAMPLES IN THE EPA-NBS TRACEABILITY STUDIES

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

AIF-NBS Quality Assurance Program

Round IX: October, 1974

IRON-59

Instruction Sheet

I. Questionnaire

A. Please read questionnaire through before proceeding.

B. Quantify material as soon as possible after receipt.

C. Please answer questions, fill in blanks or add any information you feel might be helpful in diagnosing difficulties or be helpful to other participants.

D. A certificate will be issued upon return of questionnaire, therefore the sample can also be used as a standard reference material.

E. Please return your completed questionnaire before November 15 to the

Radioactivity Section
National Bureau of Standards
Room C-114, Rad P
Washington, D.C. 20234

F. Enclosed is a copy of a t-distribution table for your use.

II. Measurement

A. Check for radionuclidic impurities, identify and quantify.

B. Determine activity of the sample.

C. Make corrections for the impurities.

D. Enter results on questionnaire.

E. Two copies each of sections II through V of the questionnaire are enclosed in the event that more than one method was used.

#3

AIF-NBS Quality Assurance Program

Round IX: October, 1974

IRON-59

Questionnaire

I. Impurities

A. Did you identify (a) radionuclidic impurity(ies)?_____ .

B. What is (are) the impurity(ies) and the relative amount(s)?

_____ _____

_____ _____

_____ _____

_____ _____

C. How did you identify and measure the impurity(ies)? _____

_____

_____

_____

_____

_____

D. List the half-life(s) and other decay scheme parameters used in evaluating the impurity(s).

_____

_____

_____

_____

_____

E. Comments: _____

_____

_____

_____

_____

II. Calibration Results: Activity per gram of solution.

A. Microcuries per gram of solution at 1200 EST October 24

_____

B. Uncertainty in the value of the activity is _____ .

1. The standard error, $\sqrt{\frac{\Sigma(x-\bar{x})^2}{n(n-1)}}$ is _____ .

2. The 99% confidence limit, (t_{n-1}) (standard error), is _____ based on _____ measurements of/on _____ source(s).

3. The total estimated systematic error is _____ which is comprised of

_____% due to _____ and

_____% due to _____ and

_____% due to _____ and

_____% due to _____ and

_____% due to _____ .

4. How are the systematic errors combined? _____

_____

_____

5. How are the random and systematic errors combined?

III. Method of Calibration: Activity per gram of solution.

A. Describe calibration technique used. Please be explicit. Your description could be very important in the analysis of the results. Use the reverse side of this sheet if necessary.

B. Please list the decay scheme parameters (with their uncertainties) used in determining your activity value.

C. List corrections made and order of magnitude (and uncertainties).

D. Please describe in detail the technique(s) used for preparing your counting source(s).

1. How many sources did you prepare? _____ .

2. How many sources did you measure? _____ .

E. If a standard was used (for comparison or efficiency determination, etc.) please give the following information:

1. What standard was used? _____

2. Chemical form _____

3. Physical form _____

4. Accuracy statement supplied with standard _____

5. Describe your use of this standard _____

_____

_____

IV. Calibration Results. Total activity in the ampoule.

A. Microcuries in ampoule at 1200 EST October 24 _____ .

B. Uncertainty in the value of the activity is _____ .

1. The standard error is

2. The 99% confidence limit is _____ based on _____ measurements of/on _____ source(s).

3. The total estimated systematic error is _____ which is comprised of

_____% due to _____ and

_____% due to _____ and

_____% due to _____ and

_____% due to _____

4. How are the systematic errors combined? _____

_____

_____

5. How are the random and systematic errors combined?

_____

_____

_____

_____

V. Method of Calibration. Total activity in the ampoule.

A. Describe calibration technique used. Please be explicit. Your description could be very important in the analysis of the results. Use the reverse side of this sheet if necessary.

_____

_____

_____

_____

_____

_____

_____

B. Please list the decay scheme parameters (with their uncertainties) used in determining your activity value.

_____ _____ _____

_____ _____ _____

_____ _____ _____

_____ _____ _____

C. List corrections made and order of magnitude.

_____ _____

_____ _____

_____ _____

_____ _____

_____ _____

D. If a standard was used (for comparison or efficiency determination, etc.) please give the following information:

1. What standard was used? _____
2. Chemical form _____
3. Physical form _____
4. Accuracy statement supplied with standard _____
5. Describe your use of this standard _____